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ARTICLE

Interfacial Behavior and Thermostability of the TZM High Temperature Brazing Seam with Ti-based Brazing Filler Metals

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Abstract: The high-temperature vacuum brazing of Titanium-Zirconium-Molybdenum (TZM) alloys was efficiently realized while using the Ti-8.5Si, Ti-33Cr and Ti-30V-3Mo brazing filler metals, separately. The thermostability of the brazing joint interface after high temperature thermal cycle was studied by SEM, EDS, wettability test and shearing test, etc, and so as to the thermostability of the brazing process. The results show that, under the technological parameter of 1520 ºC/6 min, both Ti-8.5Si and Ti-33Cr brazing filler metals have good wettability on the surface of TZM alloy, whose wetting angle is 10° and 9°, respectively. The spreading area of Ti-8.5Si brazing filler metal is larger than that of the Ti-33Cr brazing filler metal. Under the technological parameter of 1680 ºC/8 min, the Ti-30V-3Mo brazing filler metal has a 5° wetting angle on the surface of the TZM alloy. The (Ti, Mo) solid solution is formed in the interfaces of the Ti-8.5Si/TZM and the Ti-33Cr/TZM brazing joints. The center of the Ti-8.5Si/TZM brazing seam is composed of (Ti, Mo) solid solution and Ti_5Si_3 phase. The center of the Ti-33Cr/TZM brazing seam is composed of (βTi, Cr) solid solution and the αTi +(αTi+$\alpha TiCr_2$) eutectic. The brazing seam of the Ti-30V-3Mo/TZM joint is composed of (βTi, V) solid solution and the αTi phase, and the boundary zone is composed of (Ti, Mo) solid solution. They all form solid solutions in the brazing joints to realize the metallurgical bonding between the brazing filler metals and the TZM alloys when using these three kinds of brazing filler metals. The shear strengths of the brazing joints are 135.8 MPa (Ti-8.5Si), 132 MPa (Ti-33Cr) and 131 MPa (Ti-30V-3Mo). There are no obvious intergranular infiltration and parent metal dissolution phenomena in the Ti-8.5Si/TZM and Ti-33Cr/TZM brazing joints after the 1200 ºC/60 min thermal cycle treatment, and the boundary zone is still combined by the solid solution. In the Ti-30V-3Mo/TZM brazing joint after 1550 ºC/60 min thermal cycle, there is intergranular dissolution of one-crystalline-grain-depth, no obvious parent metal dissolution is observed, and the interface maintains the combination of the solid solution. These three kinds of titanium based brazing filler metals can realize the high-temperature vacuum brazing of the TZM alloy through metallurgical bonding of the interfacial solid solution. The microstructure of the brazing joint after long time high temperature thermal cycle is stable, and the intergranular infiltration sensitively is low. The research of this subject provides theoretical and experimental guidance for the high temperature application connection of the TZM alloy.

Key words: titanium-zirconium-molybdenum (TZM) alloy; high temperature brazing; titanium based filler metal; intergranular infiltration; thermostability

Titanium-Zirconium-Molybdenum (TZM) alloy is an alloy formed by adding tittles Ti, Zr and C elements into pure Mo. The melting point of the TZM alloy is 2617 ºC, and its recrystallization temperature is 1400 ºC, higher than that of pure Mo (1100±50) ºC. The coefficient of thermal expansion (CTE) of TZM is 5.3×10^{-6} K^{-1} (20~100 ºC),

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which is much lower than the CTE of iron (Fe: 12.2×10^{-6} K^{-1}). Owing to its outstanding properties such as good corrosion resistance, excellent high temperature mechanical properties and good electrical conductivity[1-4], TZM alloy is widely used in the fields of aerospace, nuclear, electronics industries and civil industries, such as rocket engine nozzle, high temperature and molten salt reactor, heating element and reflecting screen in high temperature furnace[5, 6].

Brazing can realize the reliable joining between TZM and TZM, TZM and other materials; it is one of the important methods to extend the application of TZM and other molybdenum alloys[7-10]. In order to improve the operating temperature of TZM, high-temperature brazing must be used for connection. However, in high-temperature brazing process of TZM, the reaction between brazing filler metal and parent material was intensified, and in the boundary zone it is easy to form continuous compounds. The parent material dissolution and intergranular infiltration phenomena, will cause the embrittlement of the brazing joint, and reduce the bearing capacity of the parent material[9-11]. Intergranular infiltration becomes an important factor that impedes the TZM's reliable connection at high temperature. Song et al.[12] brazed TZM alloys using Ti-28Ni (wt%) eutectic brazing filler metal in the brazing temperature range of 1000~1160 ºC, and found that the presence of δ-Ti_2Ni intermetallic phase and crack-like structure in joints deteriorated the joining properties, which result in the formation of brittle fracture after shear test. However, the research on the high temperature brazing of TZM alloy above 1400 ºC has not been reported.

In the present study, Ti-8.5Si, Ti-33Cr and Ti-30V-3Mo brazing filler metals were developed to high temperature vacuum brazing of TZM. The high temperature thermal cycle treatment was conducted after brazing. The reaction behavior between brazing filler metal and TZM was studied, the bonding mechanism in the boundary zone was discussed, and the stability of the brazing joint after long time high temperature treatment and the intergranular infiltration sensitivity on the interface were investigated.

1 Experiment Materials and Methods

The base metal is commercial TZM plate with a thickness of 5 mm. The chemical composition (wt%) of TZM are (0.4~0.55) Ti, (0.07~0.12) Zr, (0.01~0.04) C and Mo balance.

Three kinds of brazing filler metals, including Ti-8.5Si, Ti-33Cr and Ti-30V-3Mo (wt%, nominal composition) were smelted by arc melting equipment. These brazing filler metals were cut into foils with the thickness of 0.8 mm by spark cutting. The brazing filler metals were ground on SiC grit papers and cleaned with alcohol for standby application.

The brazing experiment was done in the LZQH-30 vertical vacuum brazing furnace, and the degree of vacuum is (2~5) $\times10^{-3}$ Pa. Ti-8.5Si and Ti-33Cr filler metals brazing TZM temperature is 1520 ºC, held for 10 min. Ti-30V-3Mo filler metal brazing TZM temperature is 1680 ºC, held for 8 min.

The 0.15 g brazing filler metal was placed on the surface of the 2 mm thick TZM alloy, both of which were put into the vacuum furnace for wettability test. The wetting angle of the wetting sample was measured by JC2000D contact angle measuring instrument. The spreading morphology of the brazing filler metal on the TZM plate was photographed using Zeiss Discovery V8 stereoscopic microscope. The wetting area of the brazing filler metal on the TZM plate was measured by software V4.9.1 SP2, AxioVision SE64.

Three kinds of brazing joints were put into the vacuum again to conduct the thermal cycle treatment. The brazing joints using Ti-8.5Si and Ti-33Cr brazing filler metals were kept for 60 min for thermal insulation at 1200 ºC. The brazing joint using Ti-30V-3Mo brazing filler metal was kept for 60 min for thermal insulation at 1550 ºC. The interfacial microstructure of the brazing joint after thermal cycle treatment was examined. The thermal cycle curve is shown in Fig.1.

The brazing joints were cut from the section by spark cutting and embedded with resin powder. The embedded samples were ground on 240#, 400#, 800#, 1000#, 1200#, 1500# SiC grit papers and polished using diamond pastes to observe the microstructures of the brazing joints. Morphology of the brazing filler metal was examined by optical microscope (OM, Zeiss AxioScope) and scanning electron microscope (SEM, Phenom XL). Quantitative componential analysis of specific micro regions of the brazing filler metals was performed by an energy dispersive spectrometer (EDS, SEM, Phenom XL).

Shear strength tests were conducted at room temperature by a universal testing machine (MTS E45.105). The geometric sizes of the base materials in the TZM/TZM brazing joint were 5 mm×5 mm×5 mm and 10 mm×10 mm×5 mm. The brazing filler metal was put into the middle of base TZM alloys to form a sandwich structure, and the bonding area was 25 mm^2. For

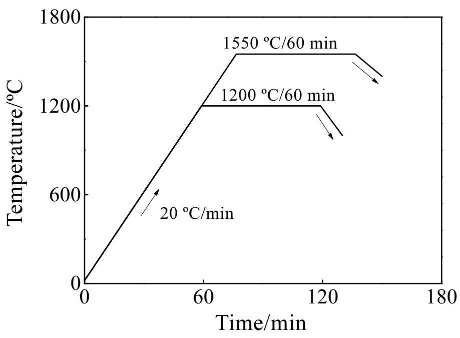

Fig.1 Thermal cycle curve of the brazing joint

each brazing condition, five shearing test specimens were used to average the lap joints strength.

2 Results and Discussion

2.1 Wettability of the brazing filler metal

The wetting angle and spreading area of Titanium based brazing filler metal on the surface of the TZM alloy are as shown in Fig.2. It can be seen from Fig.2 that, the wetting angles of Ti-8.5Si, Ti-33Cr and Ti-30V-Mo brazing filler metals on TZM plate surface are 10°, 9° and 5°, respectively. The smaller the wetting angle is, the better the wettability of brazing filler metal on TZM surface is.

Fig.2d illustrates that the spreading area of Ti-8.5Si brazing filler metal (163 mm^2) is larger than that of Ti-33Cr brazing filler metal (76 mm^2). The spreading area of Ti-30V-3Mo brazing filler metal is 153 mm^2. It is attributed to the surface of Ti-33Cr brazing filler metal consisting of Cr_2O_3 and TiO_2 phases. Under the vacuum degree ((2~5) 10^{-3} Pa) of experimental condition, the TiO_2 film on surface can be dissolved into Ti, but the stability of Cr_2O_3 is strong and cannot be completely volatilized under vacuum condition. Therefore, the presence of Cr_2O_3 film on the surface of Ti-Cr prevents the wetting and spreading of brazing filler metal on TZM alloy. In the Ti-30V-3Mo brazing filler metal, the Ti and V elements can form solid solutions with Mo element, resulting in good wettability of it on TZM plate.

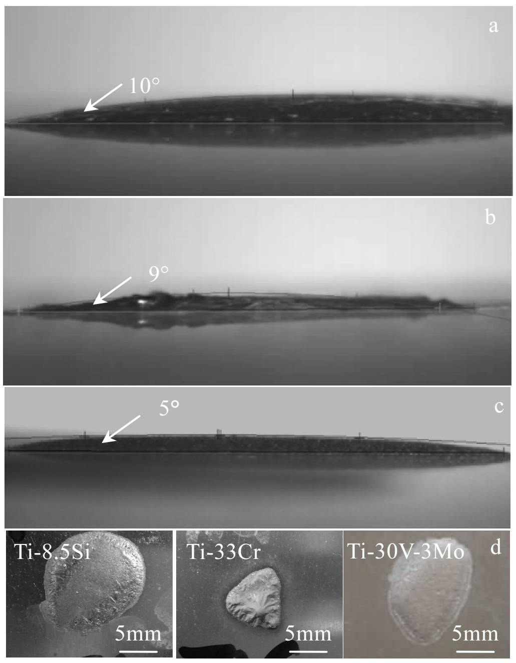

Fig.2 Wetting angles of Ti-8.5Si (a), Ti-33Cr (b), Ti-30V-3Mo (c) and spreading area (d) of Ti-based brazing filler metal on TZM surface

2.2 Interfacial microstructure of the brazing joints

Fig.3 shows the interfacial SEM image and surface EDS element mapping of the brazing joint of TZM alloy using Ti-8.5Si brazing filler metal. It can be seen from Fig.3 that the interface of the brazing joint is flat and without any obvious holes, cracks and other defects. During the brazing process, the Ti-8.5Si brazing filler metal melted, the Ti element diffused into the parent material, the Mo element diffused into the brazing seam, which were combined as (Ti, Mo) solid solution in the boundary zone. Silicon element is enriched in center of the brazing seam, to form Ti_5Si_3 phase with titanium.

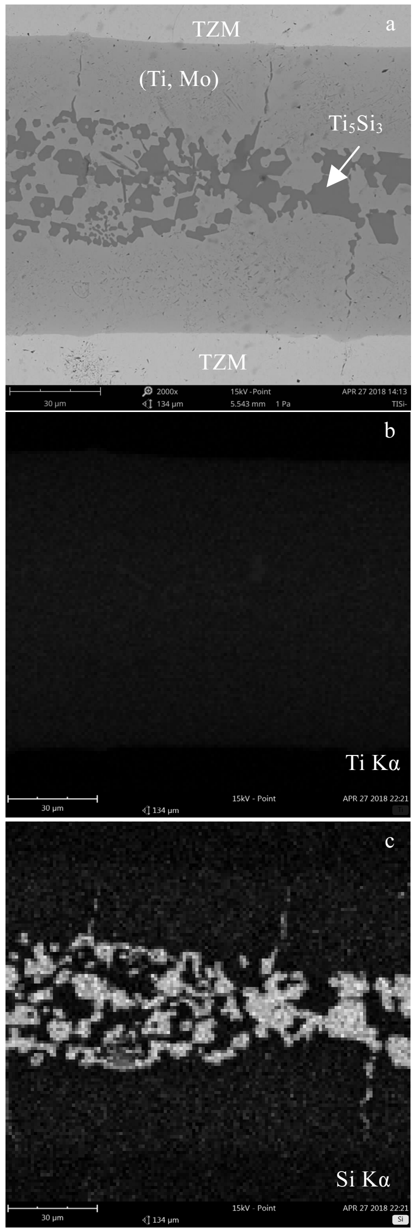

Fig.3 Interfacial SEM image (a) and surface EDS element mapping (b, c) of TZM brazing joint using Ti-8.5Si brazing filler metal: (b) Ti and (c) Si

Fig.4 shows the interfacial SEM image and surface EDS element mapping of TZM brazing joint using Ti-33Cr brazing filler metal. From Fig.4, it can be seen that Ti-33Cr brazing filler metal can realize the brazing of TZM alloy. The interface of the brazing joint is flat and without any obvious holes, cracks and other defects. The energy dispersive spectrometer (EDS) analyses of characteristic point are shown in Table 1. Combined with Fig.4, the (Ti, Mo) solid solution dissolved with Cr is formed in the boundary between the brazing filler metal and TZM alloy. In the center of the brazing seam, there occurs enrichment of Cr, Ti elements and eutectic structure (αTi+αTiCr$_2$). Between the brazing seam center and the boundary, the microstructure is composed of (βTi, Cr) solid solutions and αTi phases.

Fig.5 shows the interfacial SEM image and surface EDS element mapping of the brazing joint of TZM alloy using Ti-30V-3Mo brazing filler metal. EDS analysis of characteristic point are shown in Table 2. It can be seen from Fig.5 that, Ti-30V-3Mo brazing filler metal can realize the brazing of TZM alloy at 1680 ºC. The interface of the brazing joint is flat and without any obvious metallurgical defects, parent material dissolution and intergranular infiltration phenomena. Combined with the EDS results, the brazing seam consists of (βTi, V) solid solutions and αTi phases, while the boundary is composed of (Ti, Mo) solid solutions to realize the metallurgical bonding between brazing filler metal and TZM alloy.

2.3 Metallurgical bonding mechanism of the brazing joint

When brazing TZM alloys using Ti-8.5Si and Ti-33Cr brazing filler metals at 1520 ºC, or using Ti-30V-3Mo brazing filler metal at 1680 ºC, continuous solid solutions are formed by Ti and Mo elements to realize the metallurgical bonding between the brazing filler metals and TZM alloys. No continuous compound is formed between the brazing filler metal and TZM alloy. When brazing TZM alloy using Ti-based brazing filler metal, there are no obvious parent material dissolution and intergranular infiltration phenomena.

In the Ti-8.5Si/TZM brazing joint, the affinity between Si and Ti is greater than that of Mo and Si, Mo and Ti. Parts of Ti elements in the brazing seam interdiffuse with Mo, and parts of them react with Si elements to form compounds. The brazing filler metal does not spread to the grain boundary of TZM alloy, and no intergranular infiltration occurs. On the other hand, since Si element is enriched in the brazing seam, no Mo-Si compound is formed in the boundary. In addition, on the interface, the concentration of Ti increases, the concentration of Si decreases, continuous solid solutions are formed by Ti and Mo elements to realize the metallurgical bonding between the brazing filler metals and TZM alloys.

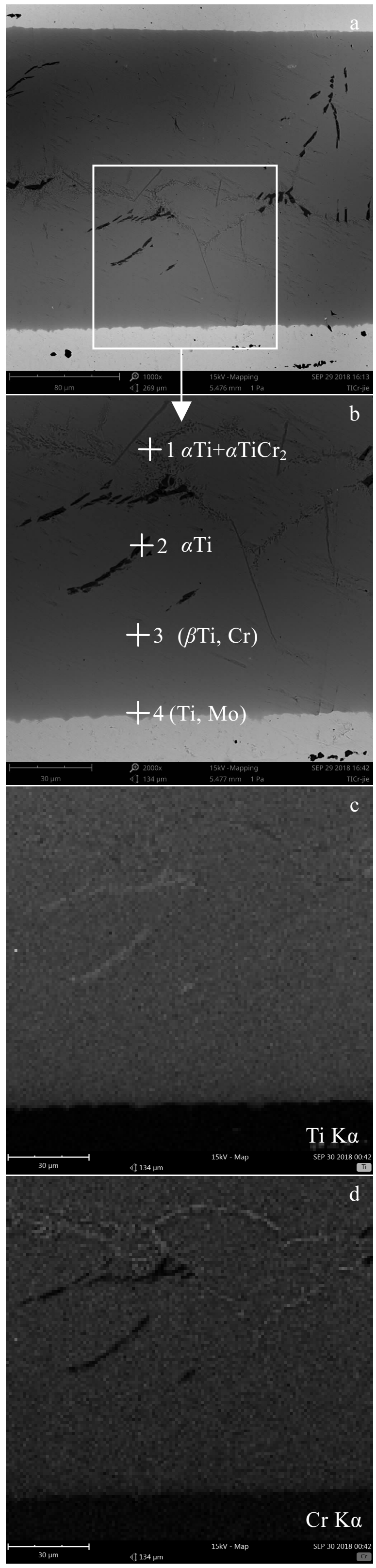

Fig.4 Interfacial SEM images (a, b) and surface EDS element mapping (c, d) of TZM brazing joint using Ti-33Cr brazing filler metal Ti (c) and Cr (d)

Table 1 EDS analysis of characteristic points of the Ti-33Cr/TZM brazing joint in Fig.4b (at%)

Point	Ti	Cr	Mo	Zr
1	60.40	36.73	2.87	-
2	97.44	1.22	1.34	-
3	66.77	21.77	10.86	0.59
4	52.77	31.29	14.72	1.22

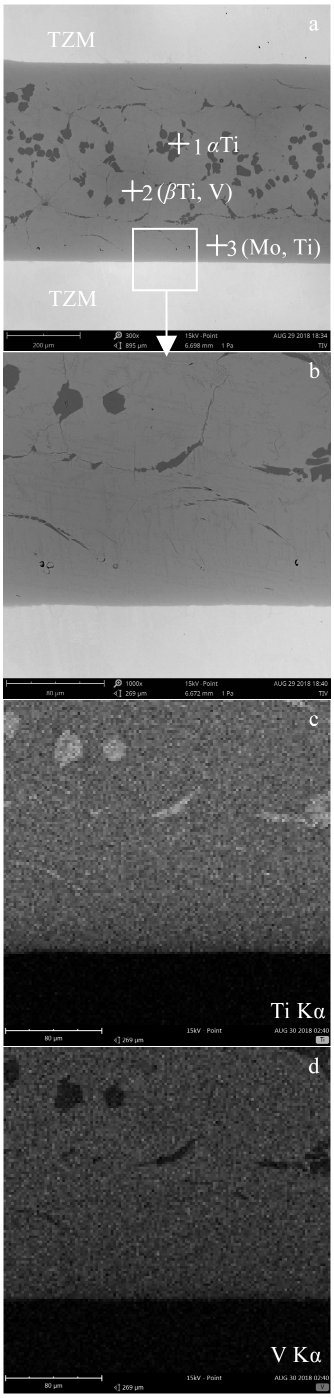

Fig.5 Interfacial SEM images (a, b) and surface EDS element mapping of TZM brazing joint using Ti-30V-3Mo brazing filler metal: (c) Ti and (d) V

In the Ti-33Cr/TZM brazing joint, Cr and Mo can react to form continuous solid solution. Cr and Ti have high solid solubility and can also form compound or eutectic structure. The affinity between Cr and Ti is greater than that of Cr and Mo. When the brazing joint solidifies, Cr is easy to combine with Ti. Cr is enriched in center of the brazing joint, and forms (αTi+αTiCr$_2$) eutectic structure with Ti. The (Ti, Mo) solid solution dissolved with Cr is formed in the boundary. No obvious intergranular infiltration phenomenon is found, while only trace dissolution occurs at the grain boundary of parent material.

When brazing TZM alloy using Ti-30V-3Mo brazing filler metal at 1680 ºC, since continuous solid solutions can be formed between Ti and V, Ti and Mo, V and Mo, the brazing joint realizes metallurgical bonding through forming (Ti, Mo) solid solution in the boundary.

2.4 Mechanical properties of the brazing joints

Fig.6 shows the shear strength of TZM brazing joints using Ti-based brazing filler metal. The shear strength of the brazing joints is about 130 MPa. Since the brazing joints using Ti-8.5Si, Ti-33Cr and Ti-30V-Mo brazing filler metals are all bonded through (Ti, Mo) solid solutions, the bonding strength between the parent material and the brazing filler metal was manifested as the bonding strength of (Ti, Mo) solid solution, so the shear strength among these three kinds of brazing joints don't differ greatly. As can be seen from the fracture morphology shown in Fig.7, the fractures of these three kinds of brazing joints are relatively flat, without obvious plastic deformation. The fracture presents the characteristics of brittle cleavage fracture. According to the EDS analysis results of the fracture surfaces (Table 3), the main residual elements of the three fracture surfaces are Ti and Mo. Under the action of shear force, the fracture positions of these three kinds of brazing joints are the joint surfaces between the brazing filler metals and the TZM parent materials, that is the (Ti, Mo) solid solution boundary zone. Due to the slightly different components of Ti and Mo in (Ti, Mo) solid solutions, and other different elements in the solid solutions, the shear strength of these three kinds of brazing joints is not identical incompletely, but the difference is not significant.

Table 2 EDS analysis of characteristic points of the Ti-30V-3Mo/TZM brazing joint in Fig.5a (at%)

Point	Ti	V	Mo	Zr
1	95.68	3.59	0.73	-
2	63.01	30.14	6.86	-
3	27.06	6.84	64.23	1.88

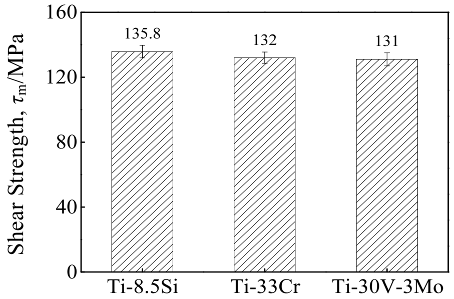

Fig.6 Shear strength of the brazing joints

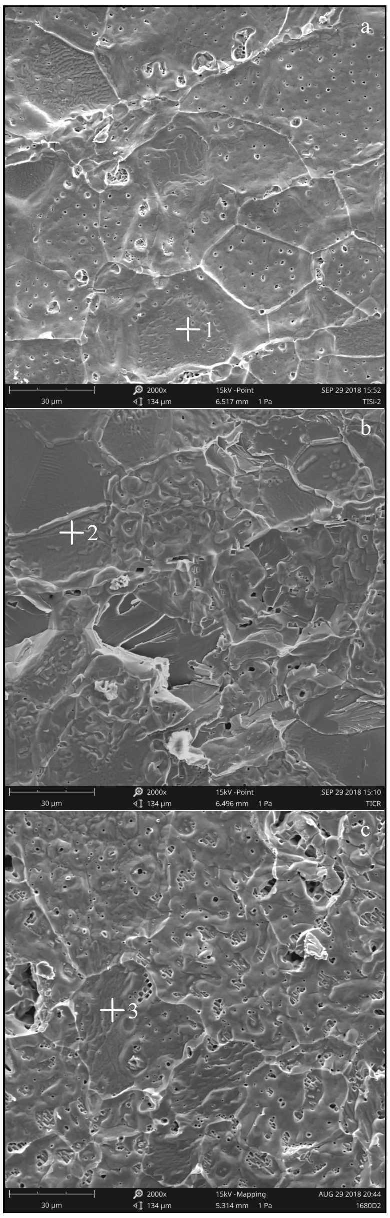

Fig.7 Shear fracture morphologies of the brazing joints: (a) Ti-8.5Si, (b) Ti-33Cr, and (c) Ti-30V-3Mo

Table 3 EDS analysis of characteristic points of the fracture surfaces of the brazing joints in Fig.7 (at%)

Point	Mo	Ti	Si	Cr	V	Zr
1	50.52	45.79	2.16	-	-	1.53
2	41.08	47.53	-	10.17	-	1.22
3	69.55	23.26	-	-	4.78	2.41

2.5 Thermostability of the brazing joints

Fig.8 shows the microstructures of TZM brazing joints after thermal cycle treatment using Ti-8.5Si, Ti-33Cr and Ti-30V-3Mo brazing filler metals. From Fig.8a and Fig.8b, the TZM brazing joints using Ti-8.5Si and Ti-33Cr brazing filler metals are combined well after 1200 ºC long time (60 min) heat preservation. There were no cracks, no obvious parent material dissolution and intergranular infiltration.

Compared with the brazing joints without thermal cycle treatment, the microstructure of the brazing joint interface and brazing seam of Ti-8.5Si/TZM do not change significantly. Mo and Ti react to form continuous (Ti, Mo) solid solution in the boundary zone, and Ti_5Si_3 phase is distributed in the brazing seam center. The brazing filler metal does not melt when the Ti-8.5Si/TZM brazing joint is undergoing thermal cycle treatment. Mo, Ti and Si elements diffuse in the brazing seam, but there is no obvious solid-state phase transformation, and the structure is basically unchanged after cooling. The microstructure of TZM brazing joint interface using Ti-33Cr brazing filler metal do not change significantly after thermal cycle treatment. Because of Mo and Ti, Cr and Mo can react to form continuous solid solutions, Ti and Cr can react to form solid solution and $TiCr_2$ compound, so at 1200 ºC thermal cycle treatment, the combination mode between the brazing filler metal and TZM alloy is unchanged. The brazing filler metal and TZM alloy is combined through (Ti, Mo) solid solution, and in center of the brazing seam, the microstructure is composed of αTi phases and (βTi, Cr) solid solutions.

It can be seen from Fig.8c that, after 60 min thermal cycle treatment at 1550 ºC, the Ti-30V-3Mo/TZM brazing joint can still maintain complete brazing seam. The brazing interface is well combined, and the (Ti, Mo) solid solution combination mode in the boundary is unchanged.

Compared with the brazing joints without thermal cycle treatment, there is only one-crystalline-grain-depth intergranular dissolution in the brazing joint boundary, which has little influence on the overall performance of the brazing joint.

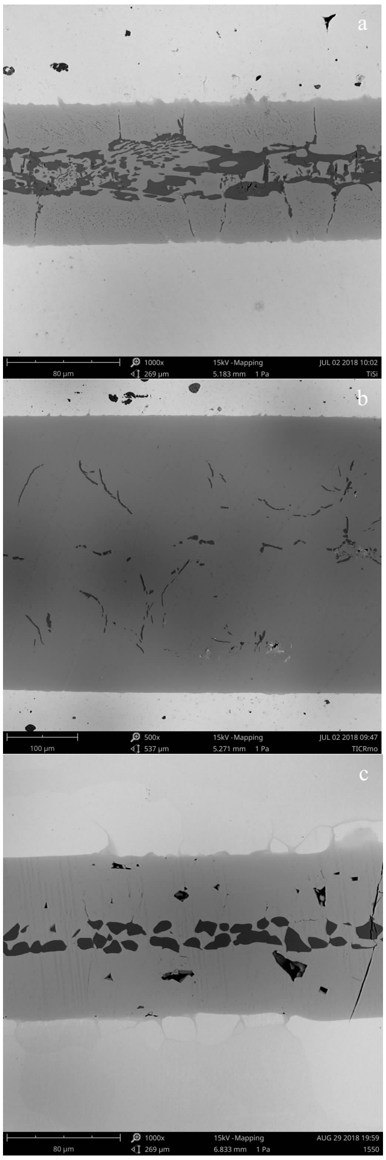

Fig.8 Microstructures of brazing joints between Ti-based brazing filler metals and TZM alloy after thermal cycle treatment: (a) Ti-8.5Si, (b) Ti-33Cr, and (c) Ti-30V-3Mo

3 Conclusions

1) Ti-8.5Si, Ti-33Cr and Ti-30V-3Mo brazing filler metals all have good wettability on TZM surface. The wetting angles of Ti-8.5Si, Ti-33Cr and Ti-30V-Mo brazing filler metals are 10°, 9° and 5°, respectively. The spreading area of Ti-8.5Si or Ti-30V-3Mo brazing filler metal is larger than that of Ti-33Cr brazing filler metal.

2) (Ti, Mo) solid solutions are formed between brazing filler metal and TZM alloy to realize the metallurgical bonding between the Ti-8.5Si, Ti-33Cr or Ti-30V-3Mo brazing filler metal and TZM alloy. No continuous compound is formed in the boundary between brazing filler metal and TZM alloy. There are no obvious intergranular infiltration and TZM parent material dissolution phenomena. The shear strength of Ti-8.5Si/TZM, Ti-33Cr/TZM and Ti-30V-3Mo/TZM brazing joints are 135.8, 132 and 131 MPa, respectively. Because of the solid solution interface bonding mechanism between these three brazing filler metals and TZM alloys, the shear strength among these three kinds of brazing joints do not differ greatly.

3) There are no obvious intergranular infiltration and parent material dissolution in the 8.5Si/TZM and Ti-33Cr/TZM brazing joints after the 1200 ºC/60 min thermal cycle treatment, and the boundary zone is still combined by the solid solution. In the Ti-30V-3Mo/TZM brazing joint after 1550 ºC/60 min thermal cycle, there is one-crystalline-grain-depth intergranular dissolution, and no obvious parent metal dissolution is observed, and the interface remains combined by the solid solution. These three kinds of Titanium based brazing filler metals can realize the high-temperature vacuum brazing of the TZM alloy through metallurgical bonding of the interfacial solid solution. The microstructure of the brazing joint after long time high temperature thermal cycle is stable, and the intergranular infiltration sensitively is low.

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钛基钎料高温钎焊 TZM 界面行为及热稳定性研究

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摘　要：采用Ti-8.5Si、Ti-33Cr和Ti-30V-3Mo钎料实现了钛锆钼（TZM）合金的高温真空钎焊连接，借助SEM、EDS及润湿性试验和抗剪试验等分析方法，研究了钛基钎料高温钎焊TZM及钎焊接头经高温热循环后的热稳定性。结果表明，Ti-8.5Si、Ti-33Cr在1520 ºC/6 min的工艺条件下良好润湿TZM，润湿角分别为10º和9º，Ti-8.5Si钎料的铺展面积大于Ti-33Cr钎料的铺展面积，Ti-30V-3Mo钎料在1680 ºC/8 min的条件下在TZM板上的润湿角为5°。Ti-8.5Si/TZM接头界面形成（Ti, Mo）固溶体，钎缝中心由（Ti, Mo）固溶体和Ti_5Si_3相组成。Ti-33Cr/TZM接头界面形成（Ti, Mo）固溶体，钎缝中心由（βTi, Cr）固溶体和αTi+(αTi+$\alpha TiCr_2$)共晶组成。Ti-30V-3Mo/TZM接头，钎缝区主要由（βTi, V）固溶体和αTi组成，界面区由Ti与Mo形成（Ti，Mo）固溶体。3种钎料钎焊TZM，均形成固溶体钎焊接头而实现钎料与TZM的冶金结合，钎焊接头强度分别为135.8 MPa（Ti-8.5Si）、132 MPa（Ti-33Cr）和131 MPa（Ti-30V-3Mo）。Ti-8.5Si/TZM、Ti-33Cr/TZM接头经过1200 ºC/60 min没有观察到明显的晶间渗入和母材溶蚀，界面固溶体结合形式无变化。Ti-30V-3Mo/TZM接头经过1550 ºC/60 min热循环后，观察到1个晶粒深度的晶间腐蚀，没有明显的母材溶蚀现象，且界面依然保持固溶体结合形式。3种钛基钎料可实现TZM的高温钎焊，依靠界面固溶体实现冶金结合，经高温长时间热循环后钎焊接头组织性能稳定，发生晶间渗入敏感性低，为TZM的高温应用连接提供理论与试验指导。

关键词：钛锆钼合金；高温钎焊；钛基钎料；晶间渗入；热稳定性

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